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INVENTORS:

**CARL R. VANDER LINDEN; JAMES P.  
LEINEWEBER**

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OWNERS:

**JOHNS-MANVILLE CORPORATION**

ISSUED:

**Dec. 11, 1962**

FILED:

LAID OPEN:

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**23/186**

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ABSTRACT:

CLAIMS: [Show all claims](#)

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- 2 -

053797

1 This invention relates to improved hydrated calcium  
2 silicate products and their preparation. More particularly,  
3 the invention is concerned with an improved method of  
4 buffering the basic characteristics of hydrated calcium  
5 silicates and products thereof.

6 Particulate hydrated calcium silicates have for some  
7 time been proposed for use as fillers, pigments, extenders,  
8 etc., for paint, paper, rubber, plastic, and the like pro-  
9 ducts and in some applications such silicates have been  
10 found effective to varying degrees. However, many calcium  
11 silicate compositions now available typically exhibit one  
12 or more properties such, for example, as relatively high  
13 solubilities, high alkalinity or basic pH, among others,  
14 which render them unsatisfactory and/or impractical, and  
15 frequently inoperative in many applications or products  
16 wherein a substantially completely insoluble or "inert" and/  
17 or approximately neutral or even acid filler or pigment  
18 possessing other properties not unlike those of the highly  
19 absorptive calcium silicates could effectively and economic-  
20 ally fulfill many filler, pigment or extender requirements  
21 in the paint, paper, rubber, plastic and allied industries.

22 Efforts to overcome various of the noteworthy dis-  
23 advantages of the more common hydrated calcium silicate  
24 products in particular applications and thereby extend their  
25 scope of utility have to date been met with little success.  
26 For example, pre-treatment of highly basic hydrated calcium  
27 silicate compounds by contacting the same with an acid  
28 material such as alum, sulfuric acid or the like, has hereto-  
29 fore been proposed as a means of rendering calcium silicates  
30 suitable for use in products or processes which involve or  
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1 require a low or acid pH medium. Such a pre-treatment of the  
2 conventional hydrated calcium silicates, however, has proven  
3 to be relatively ineffective and/or uneconomical in that  
4 amounts of acid material approximately sufficient to decom-  
5 pose or react with substantially all of the calcium silicate  
6 product are typically required to effectively reduce the pH  
7 of an aqueous slurry of the silicate to about 4 - 5. In  
8 other words, substantially stoichiometric quantities of an  
9 acid material are required to materially reduce the high pH  
10 values of an aqueous suspension of calcium silicate and  
11 maintain the same at a low pH value. Thus, a partial pre-  
12 treatment of a calcium silicate with amounts of an acid  
13 material lacking stoichiometric proportions, in preparing  
14 the same for application in a process or product requiring  
15 an acid medium such as paper-making slurries, typically  
16 results in a product which, if desirable or essential to  
17 maintain at a low or acid pH, requires a subsequent addi-  
18 tion(s) of substantial proportions of an acid-imparting  
19 material in amounts which when totaled with the pre-treatment  
20 acid component approaches uneconomical stoichiometric pro-  
21 portions.

22 It is an object of this invention to provide an  
23 improved method of preparing alum treated hydrated calcium  
24 silicates and the products thereof.

25 It is also an object of this invention to provide an  
26 effective and economical method of buffering the basic  
27 characteristics of stable high temperature phase hydrated  
28 calcium silicate products.  
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- 4 -

053797

1 It is a further object of this invention to modify the  
2 surface characteristics of slow filtering high temperature  
3 phase hydrated calcium silicates to materially improve their  
4 filtering rates.

5 It is a still further object of this invention to pro-  
6 vide stable high temperature phase hydrated calcium silicate  
7 products exhibiting relatively low pH characteristics which,  
8 among other advantages and potential applications, comprises  
9 effective and economical fillers, pigments, and/or extenders  
10 for paint, paper, rubber, plastic, and the like products.

11 This invention will be more fully understood and fur-  
12 ther advantages and objects thereof will become apparent  
13 from the hereinafter more detailed description and specific  
14 examples taken in connection with the accompanying drawings,  
15 in which:

16 Fig. 1 illustrates the relative effectiveness of the  
17 treatment of this invention and the pH characteristics of  
18 the products of said treatment in comparison with those of  
19 procedures not within the scope of this invention;

20 Fig. 2 illustrates the unreceptiveness of low tempera-  
21 ture phase hydrated calcium silicate products which as cal-  
22 cium silicate hydrate I to all types of treatment including  
23 the novel method of this invention;

24 Fig. 3 compares the relative effectiveness of the  
25 method of this invention as applied to various high tempera-  
26 ture phase hydrated calcium silicate compositions; and

27 Fig. 4 illustrates the long term alum stability of  
28 products treated in accordance with this invention.  
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- 5 -

653797

1 Typical hydrated calcium silicate products when sub-  
2 jected to an aqueous acid medium in the performance of a  
3 process or in the preparation of a product, for example a  
4 paper-making furnish wherein alum (aluminum sulfate) is  
5 often added to maintain the pH of the furnish within the  
6 approximate range of 4 - 6, react with the acidic component(s)  
7 of said medium with a resultant overall increase in pH.  
8 Thus, whenever essential, or even desirable to maintain or  
9 restore the low pH condition of the medium, an additional  
10 amount of the acidic or acid producing component must be  
11 added thereto to restore or maintain the pH at the desired  
12 or original level. Such a practice is often costly and  
13 therefore prohibitive in many manufacturing procedures or  
14 products.

15 This invention provides new hydrated calcium silicates  
16 exhibiting relatively low pH characteristics, among other  
17 advantageous properties, which are peculiarly adaptable for  
18 use as fillers, pigments, extenders, etc., in products or  
19 processes involving or necessitating relatively low or acid  
20 pH mediums. The novel silicate products of this invention  
21 are produced by treating or reacting stable, high temperature  
22 phase hydrated calcium silicate compounds with aluminum  
23 sulfate at temperatures of at least about 350°F. and prefer-  
24 ably within the approximate range of 450° to 550°F. A  
25 temperature of about 450°F. is most preferred for reasons of  
26 efficiency and economy. Further, the treatment or reaction  
27 should comprise sufficient aluminum sulfate and be permitted  
28 to proceed to a point wherein at least 5% by weight of the  
29 calcium oxide component of the particular silicate has  
30 reacted with aluminum sulfate, it being understood that the  
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- 6 -

853797

1 theoretical stoichiometric proportions of aluminum sulfate  
2 and calcium oxide comprise 1 mol of aluminum sulfate per 3  
3 mols of calcium oxide. The degree to which it is desirable  
4 or appropriate to extend the aluminum sulfate treatment  
5 beyond the foregoing stated 5% minimum, however, depends  
6 upon the requirements desired of the ultimate product.

7 As stated hereinbefore the high temperature aluminum  
8 sulfate treatment of this invention is only effective when  
9 applied to high temperature phase hydrated calcium silicate  
10 compounds, viz., hydrothermal reaction products of an aqueous  
11 suspension of lime and a reactive siliceous material, such as  
12 diatomaceous earth, quartz, etc., at temperature of at least  
13 about 370°F. and preferably approximately 450°F. Exemplary  
14 of suitable high temperature phase calcium silicate compounds  
15 are the calcium silicate xonotlite ( $5\text{CaO} \cdot 5\text{SiO}_2 \cdot \text{H}_2\text{O}$ ) and a  
16 very low solubility calcium silicate compound having the  
17 formula  $2\text{CaO} \cdot 3\text{SiO}_2 \cdot 1-2.5\text{H}_2\text{O}$  and a distinguishing X-ray diff-  
18 raction pattern having very strong lines  $d = 3.12\text{\AA}$  and  $d =$   
19  $4.12\text{\AA}$  and a medium line at  $d = 8.34\text{\AA}$  described in Canadian  
20 Patent No. 601,158, issued July 5, 1960. The foregoing high  
21 temperature phase compounds may be prepared by the hydro-  
22 thermal reaction of lime and a source of reactive silica in  
23 the respective mol ratios of 1 mol of CaO per mol of  $\text{SiO}_2$  and  
24 0.5 - 0.7 mols of CaO per mol of  $\text{SiO}_2$  in aqueous suspension  
25 at temperatures of at least about 370°F., preferably 450°F.,  
26 for periods typically of about 2 hours. By varying the mol  
27 ratios of the reactive lime and siliceous components calcium  
28 silicate products comprising mixtures of the various stable,  
29 high temperature phase hydrated calcium silicates may be pro-  
30 duced and such mixtures are likewise applicable in the  
31 practice of this invention.

- 7 -

653797

1 The extent to which the foregoing aluminum sulfate  
2 treatment of the hydrated calcium silicate may be effected  
3 depends, naturally, upon the relative proportions or mol  
4 ratios of aluminum sulfate added to the calcium silicate  
5 compound and as such can range from unaccountable stoichio-  
6 metric proportions, i.e., substantially complete or total  
7 reaction or consumption of all the available calcium oxide  
8 component of the calcium silicate consisting of 1 mol of  
9  $Al_2(SO_4)_3$  per 3 mols of  $CaO$ , down through any proportions or  
10 percentages thereof to the lowest effective limit of at  
11 least about 5% of the calcium oxide content of the particu-  
12 lar calcium silicate compound. Suitable aluminum sulfate  
13 treated calcium silicate products for many applications  
14 comprise those resulting from a treatment with sufficient  
15 aluminum sulfate to react with approximately 7-1/2% of the  
16 available calcium oxide of the particular silicate (i.e.,  
17 about 0.025 mols  $Al_2(SO_4)_3$  per mol of  $CaO$ ).

18 The effectiveness of the foregoing aluminum sulfate  
19 treatments as well as that of previous methods, i.e., the  
20 extent or degree to which said treatments reduce or suppress  
21 the basic pH characteristics of the treated calcium silicate  
22 product, may be measured by the amount of aluminum sulfate  
23 required either to reduce a slurry of a given amount of an  
24 aluminum sulfate treated calcium silicate to a predetermined  
25 pH, or to maintain a predetermined maximum pH for a slurry  
26 or aqueous medium following the addition thereto of an  
27 aluminum sulfate treated calcium silicate. The effectiveness  
28 or extent which the treatment reduces or suppresses the  
29 basic pH characteristics of the treated calcium silicate and  
30 appropriate means for measuring the same are referred to  
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- 8 -

853797

1 hereinafter as the "alum demand" of the aluminum sulfate  
2 treated or buffered calcium silicate. The specific test  
3 utilized in determining the "alum demand" of the treated  
4 calcium silicates in the following examples and throughout  
5 the specification, unless indicated otherwise, comprises  
6 adding to a 1/2 gram sample of the particular aluminum  
7 sulfate treated calcium silicate slurried in 400 ml. of  
8 water, 1 ml. increments of aluminum sulfate solution con-  
9 taining 0.0216 grams of hydrated aluminum sulfate ( $Al_2(SO_4)_3 \cdot$   
10  $18H_2O$ ) per ml. and determining the pH after stirring for 5  
11 minutes. The "alum demand" is expressed as the mls. of  
12 aluminum sulfate solution which must be added to reduce the  
13 pH of the slurry to 5.0.

14 The aluminum sulfate treatment of stable, high tempera-  
15 ture phase hydrothermally prepared hydrated calcium silicate  
16 compounds may be effected in substantially any convenient or  
17 appropriate manner it being essential only that the high  
18 temperature phase hydrated calcium silicate and aluminum  
19 sulfates are reacted in an aqueous medium at temperatures of  
20 at least about 350°F. and preferably approximately 450°F.  
21 Moreover, the aluminum sulfate treatment may be practically  
22 and economically carried out in the same reactor vessel or  
23 chamber utilized to synthesize the high temperature calcium  
24 silicate simply by adding an aqueous solution of aluminum  
25 sulfate directly to said reactor upon substantial completion  
26 of the hydrothermal formation of the hydrated calcium sili-  
27 cate product and maintenance of the synthesizing temperatures.  
28 Such a procedure, addition of the aluminum sulfate directly  
29 to the hot reactor vessel contents, provides a substantial  
30 savings over subsequent heating of all components to a suit-  
31 able reaction temperature.  
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- 9 -

053797

1 The aluminum sulfate reagent suitable for carrying the  
2 invention into effect may comprise ordinary "paper-maker's  
3 alum" or any of the commercial grades of aluminum sulfate  
4 available on the market.

5 The mechanism of this invention being somewhat problem-  
6 atical, the following theoretical explanation is given for  
7 purposes of illustration rather than limitation. However,  
8 extensive observations indicate that the treatment of  
9 stable, high temperature phase hydrated calcium silicates  
10 with aluminum sulfate at the specified necessary temperatures  
11 results in a reaction product(s) of the aluminum sulfate  
12 and calcium silicate forming a protective coating on or  
13 modification of the surfaces of the hydrated calcium silicate  
14 particles providing the same with effective resistance from  
15 further acid attack.

16 The following examples illustrate the present invention  
17 including several variations in the practice of the same,  
18 and compare the invention with certain known prior art  
19 practices and other procedures outside the scope of this  
20 invention. It is to be understood that the hereinafter  
21 examples are given for purposes of illustration rather than  
22 limitation and that the specified techniques or procedures  
23 set forth are merely exemplary and are not to be construed  
24 to limit the invention to any particular means of practicing  
25 the same.

26 These examples illustrate the hydrothermal preparation  
27 of a suitable high temperature phase hydrated calcium sili-  
28 cate compound coupled with the subsequent aluminum sulfate  
29 treatment which comprises inventive subject matter of this  
30 application.

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- 10 -

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EXAMPLE I

1  
2 Eighty-five lbs. of diatomaceous earth suspended in 60  
3 gals. of water were charged to a reactor, steam pre-heated  
4 to a temperature of 450°F. and drained of condensate. Upon  
5 return of the reactor temperature to 450°F., 100 lbs. of  
6 hydrated lime in 60 gals. of water (giving a calculated CaO/  
7 SiO<sub>2</sub> mol ratio of approximately 1.0) was added thereto and  
8 the temperature again raised to 450°F. and maintained there  
9 for about 1-1/2 hours. Finally, 18-1/2 lbs. of paper-maker's  
10 alum in 40 gals. of water (0.024 moles of hydrated aluminum  
11 sulfate per mol of CaO) was added to the reactor and main-  
12 tained therein for approximately 1/2 hour for a total  
13 reaction time of 2 hours at a temperature of 450°F. The  
14 product was then drained into a holding tank and filtered  
15 over a rotary vacuum filter, oven dried and pulverized.

EXAMPLE II

16  
17 An aluminum sulfate treated xonotlite calcium silicate  
18 was prepared by maintaining an aqueous suspension of 1800  
19 gals. of diatomaceous earth slurry comprising 0.63 lbs. of  
20 diatomite per gal. and about 680 gals. of lime slurry com-  
21 prising 1.46 lbs. of CaO per gal. in a reaction vessel for  
22 1-1/2 hours at a temperature of about 460°F. The relative  
23 proportions of lime and siliceous components thereof were  
24 calculated to give a CaO/SiO<sub>2</sub> mol ratio of 1.0. Upon com-  
25 pletion of the reaction comprising 1-1/2 hours at tempera-  
26 tures of about 460°F., an aqueous solution of aluminum  
27 sulfate comprising 2.0 lbs. per gal. was added to the react-  
28 ion vessel until the aluminum sulfate content thereof reached  
29 2.4 lbs. per lb. of CaO component of the calcium silicate  
30 (about 0.20 moles of hydrated aluminum sulfate per mol of CaO)

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- 11 -

653797

1 and the combined reaction mixture was maintained at a temp-  
2 erature of approximately 460°F. for an additional period of  
3 about 30 minutes. The contents of the reactor were then  
4 filtered, dried and ground.

#### 5 EXAMPLE III

6 A suitable high pressure reactor vessel was first pre-  
7 heated with steam to a temperature of approximately 450°F.  
8 and upon draining of the condensate was charged with 85 lbs.  
9 of diatomaceous earth suspended in 60 gals. of water and  
10 the temperature was brought back to 450°F. One hundred lbs.  
11 of hydrated lime, also suspended in 60 gals. of water, was  
12 then charged to the reactor and the temperature again raised  
13 to 450°F. and held there for a 2-hour reaction period. The  
14 relative proportion of lime and siliceous material was cal-  
15 culated to give a  $\text{CaO/SiO}_2$  mol ratio of 1.0. Upon completion  
16 of the reaction period the reaction product was drained into  
17 a holding tank and the total solids in the reactor slurry  
18 were determined by evaporating a known volume of the slurry  
19 to dryness to calculate the hydrated calcium silicate content  
20 thereof for subsequent treatment. Sufficient aluminum sul-  
21 fate to react with about 15% of the  $\text{CaO}$  content of the  
22 calcium silicate (0.237 lbs. of hydrated aluminum sulfate  
23 per lb. of hydrated calcium silicate) was dissolved in about  
24 20 gals. of water and added to the aqueous suspension of  
25 hydrated calcium silicate in the holding tank with continu-  
26 ous stirring and the contents thereof maintained at a tempera-  
27 ture of about 160°F. for 1 hour. This addition of aluminum  
28 sulfate treating agent caused the slurry to thicken consid-  
29 erably and it was necessary to add additional water, about  
30 half the initial slurry volume, to keep the slurry in  
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- 12 -

653797

workable condition. The treated product was filtered over a rotary vacuum filter, dried at 240°F. in an oven and ground.

The "alum demand" of the products of Examples I and II, comprising a 7-1/2% and a 60% aluminum sulfate treated xonotlite in accordance with this invention, Example III, comprising a 7-1/2% conventionally treated xonotlite, and an untreated xonotlite as a standard were determined in accordance with the foregoing stated procedure, i.e., titrating 1 ml. increments of aluminum sulfate solution containing 0.0216 grams of hydrated aluminum sulfate ( $Al_2(SO_4)_3 \cdot 18H_2O$ ) per ml. against a 1/2 gram sample of each of the specified aluminum sulfate treated calcium silicate products and the untreated xonotlite slurried in 400 mls. of water and determining the pH after stirring for 5 minutes. The results of the foregoing test were plotted on a graph for comparison of the "alum demand" of the untreated calcium silicate xonotlite and those treated according to prior practices. These results comprise the graph of Fig. 1. The "alum demand" is expressed as the mls. of aluminum sulfate solution which must be added to reduce the pH of the slurry to 5.0.

#### EXAMPLE IV

Three identical samples of a low temperature phase hydrated calcium silicate compound identified in the art as the phase calcium silicate hydrate I (Taylor, Journal of the Chemical Society, 163, 1953) were prepared by reacting hydrated lime and a siliceous material in a mol ratio of 0.6 CaO to 1 SiO<sub>2</sub> at a temperature of 350-360°F. for a period of about 2 hours. Sample 1 was retained untreated as a standard. The second sample was treated with sufficient aluminum sulfate to react with 7-1/2% of the lime content thereof at

- 13 -

653797

1 ambient temperatures. The third sample was retained in the  
2 reactor and treated with sufficient aluminum sulfate to  
3 react with 7-1/2% of the lime content thereof at tempera-  
4 tures in the vicinity of 350 - 360°F. The "alum demand" of  
5 each sample, determined exactly in accordance with the fore-  
6 going procedure, was plotted for comparison and comprises  
7 the graph of Fig. 2 of the drawing. This experiment accord-  
8 ingly demonstrates that only the higher temperature (above  
9 about 370°F., preferably approximately 450°F.) phase calcium  
10 silicates can be made resistant to acidic conditions by  
11 treatment with aluminum sulfate.

#### 12 EXAMPLE V

13 A series of hydrothermal synthesis of high temperature  
14 phase hydrated calcium silicate products comprising reacting  
15 lime and siliceous reactants in CaO/SiO<sub>2</sub> mol ratios varying  
16 progressively from 0.5 to 1.0 CaO to 1 SiO<sub>2</sub> at temperatures  
17 of about 450°F. for a period of about 1-1/2 hours was  
18 effected. Each of the resulting hydrated calcium silicate  
19 products thereof, comprising either xenotlite, the low  
20 solubility calcium silicate having the formula 2CaO.3SiO<sub>2</sub>.  
21 1-2.5H<sub>2</sub>O referred to hereinbefore or mixtures of the said  
22 compounds, were treated by adding sufficient aluminum sul-  
23 fate to the reactor vessels to react with 7-1/2% of the  
24 calcium oxide content of the specific compound and continuing  
25 the reactions for periods of about 1/2 hour and at tempera-  
26 tures of approximately 450°F. The decreasing CaO/SiO<sub>2</sub> mol.  
27 ratios of the hydrated calcium silicate resulted in only a  
28 very slight increase in "alum demand". The "alum demand" of  
29 each of these products, determined in accordance with the  
30 foregoing test, is plotted in the graph of Fig. 3.

- 14 -

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EXAMPLE VI

Six identical samples of the high temperature phase calcium silicate xonotlite were prepared in accordance with the hydrothermal procedure and aluminum sulfate treatment of Example II. The respective xonotlite samples were treated with aluminum sulfate to varying degrees by maintaining each in an aqueous medium for a period of about 1/2 hour at a temperature of approximately 450°F. with sufficient aluminum sulfate reagent to react with about 7-1/2%, 30%, 40%, 50%, 60% and 90% of their CaO content. The long term "alum demands" for each of the thus prepared aluminum sulfate treated calcium sulfate samples was determined for comparison and evaluation by slurrying 0.50 grams of each of said samples in 400 ml. of water, adding aluminum sulfate to each slurried sample in amount equivalent to 0.086 lbs. per lb. of calcium silicate and measuring and recording the pH of each slurry sample as a function of time. The results of these tests, shown in Fig. 4, illustrate the lasting low pH properties imparted by the 60% treatment. Because of the large differences in the stability of these samples, the time is plotted on a logarithmic scale.

EXAMPLE VII

A low solubility high temperature phase hydrated calcium silicate having the formula  $2\text{CaO} \cdot 3\text{SiO}_2 \cdot 1-2.5\text{H}_2\text{O}$  was treated with 21.8 lbs. of paper-maker's alum ( $\text{Al}_2(\text{SO}_4)_3 \cdot 16\text{H}_2\text{O}$ ) per 100 lbs. of the lime component in a hydrothermal reaction vessel for a period of 30 minutes at a temperature of about 450°F. This is sufficient aluminum sulfate to react with approximately 7-1/2% of the CaO content of the calcium silicate. A comparison of the filtration rate of

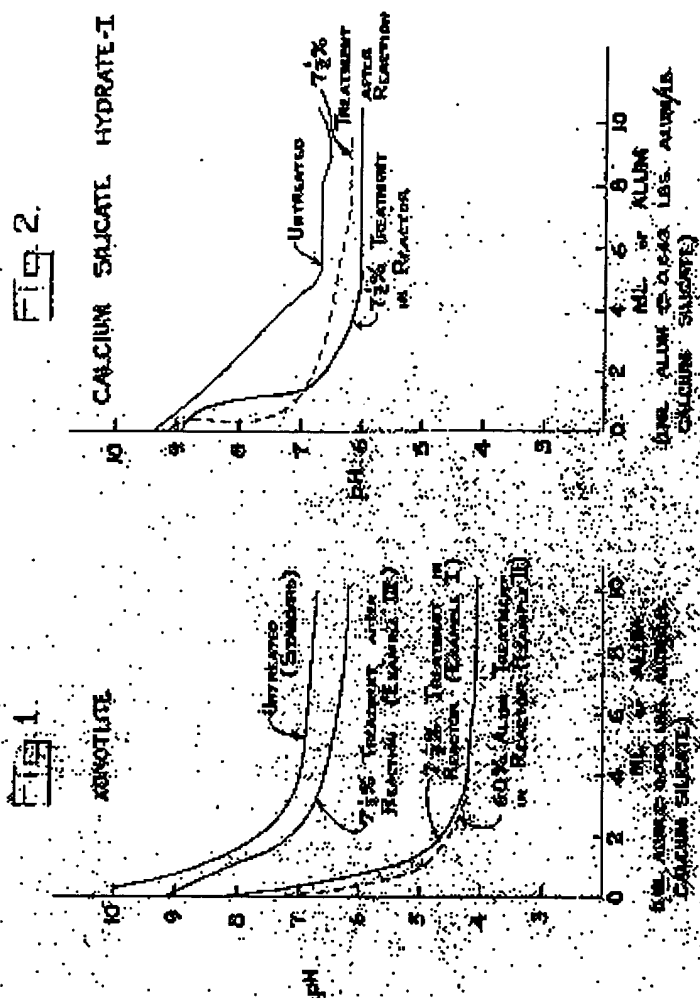
- 15 -

053797

1 the foregoing alum treated hydrated calcium silicate was  
2 made with an identical untreated low solubility high tempera-  
3 ture phase hydrated calcium silicate compound. Under identi-  
4 cal conditions the filtration rate of the aluminum sulfate  
5 treated product was 17.4 lbs. per hour per sq. ft. whereas  
6 the untreated product gave a filtration rate of 7.95 lbs.  
7 per hour per sq. ft.

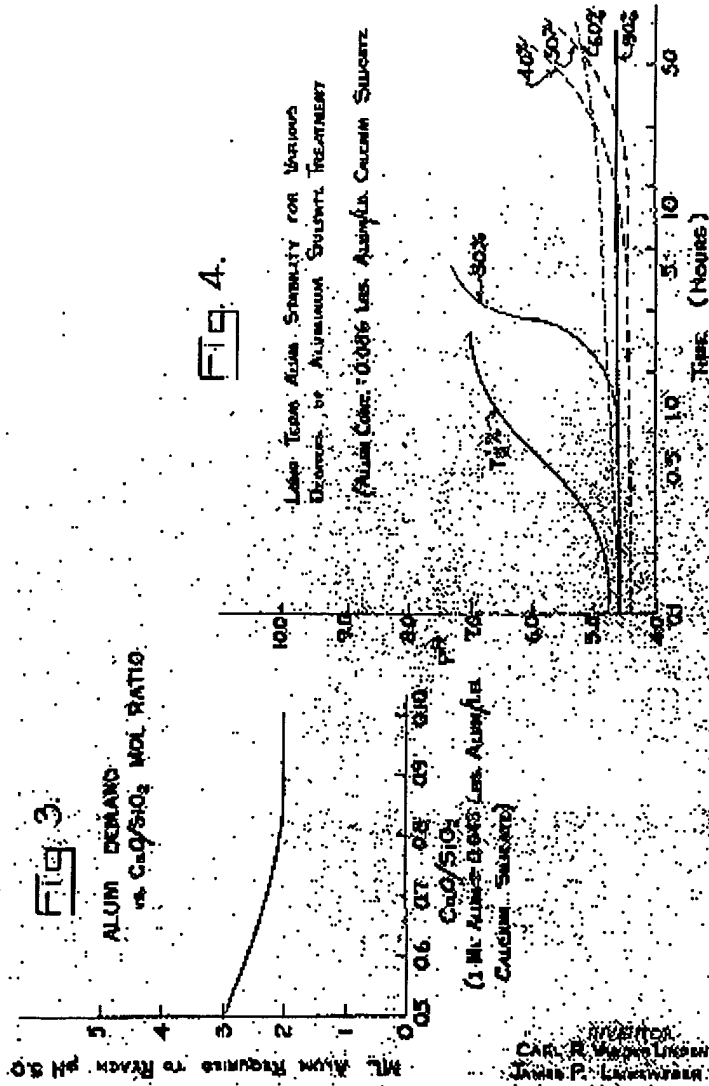
#### 8 EXAMPLE VIII

9 Several 5 gal. pressure reactors were each charged with  
10 438 grams of diatomaceous earth, 151 grams of hydrated lime  
11 and 3-1/2 gals. of water to provide a  $\text{CaO/SiO}_2$  mol ratio of  
12 1.0, and upon completion of each 90-minute reaction period  
13 at 450°F. producing the stable high temperature phase  
14 hydrated calcium silicate xonotlite, aluminum sulfate was  
15 added to each reactor in 1/2 gal. of water and after an  
16 additional 1/2 hour reaction at 450°F. the product was drain-  
17 ed from each reactor, filtered, dried and ground. The  
18 aluminum sulfate treatments were carried out at theoretical  
19 levels of 3, 6 and 30% of the amount of aluminum sulfate  
20 required to react with the CaO content of the calcium sili-  
21 cate (a 100% aluminum sulfate treatment requiring 1 mol of  
22 aluminum sulfate per 3 mols of calcium oxide of the calcium  
23 silicate product). When about 6% or greater aluminum sulfate  
24 was used, the final "alum demand" of the product was found  
25 to be less than 0.14 lbs. aluminum sulfate per lb. of calcium  
26 silicate, at the 3% level of treatment the "alum demand" was  
27 considerably higher.  
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33



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653797

The embodiments of the invention in which an exclusive property or privilege is claimed are defined as follows:

1. An improved method of preparing an aluminum sulfate treated, particulate hydrated calcium silicate product comprising hydrothermally reacting at a temperature of at least about 350°F. stable, hydrothermally formed, high temperature phase hydrated calcium silicates with aluminum sulfate in proportions of at least 0.0166 mols of aluminum sulfate per mol of CaO to effect reaction of at least 5% by weight of the total CaO component of the hydrated calcium silicates with the aluminum sulfate.
2. An improved method of preparing an aluminum sulfate treated, particulate hydrated calcium silicate product comprising hydrothermally reacting at a temperature of at least about 350°F. stable, hydrothermally formed, high temperature phase hydrated calcium silicates with aluminum sulfate in proportions of at least approximately 0.025 mols of aluminum sulfate per mol of CaO to effect reaction of approximately 7½ to 60% by weight of the total CaO component of the hydrated calcium silicates with the aluminum sulfate.
3. An improved method of preparing an aluminum sulfate treated, particulate hydrated calcium silicate product, comprising hydrothermally reacting at a temperature of approximately 450°F. stable, hydrothermally formed, high temperature phase hydrated calcium silicates with aluminum sulfate in proportions of at least approximately 0.0166 mols of aluminum sulfate per mol of CaO to effect reaction of at least 5% by weight of the total CaO component of the hydrated calcium silicates with the aluminum sulfate.

653797

4. An improved method of preparing an aluminum sulfate treated, particulate hydrated calcium silicate product comprising hydrothermally reacting at a temperature of approximately 450°F. stable, hydrothermally formed, high temperature phase hydrated calcium silicates with aluminum sulfate in proportions of at least approximately 0.025 mols of aluminum sulfate per mol of CaO to effect reaction of approximately 7½ to 60% by weight of the total CaO component of the hydrated calcium silicates with the aluminum sulfate.

5. An improved method of preparing an aluminum sulfate treated, particulate hydrated calcium silicate product comprising hydrothermally reacting at a temperature of approximately 450°F. stable, hydrothermally formed, high temperature phase hydrated calcium silicates with aluminum sulfate in proportions of at least approximately 0.025 mols of aluminum sulfate per mol of CaO to effect reaction of approximately 7½% by weight of the total CaO component of the hydrated calcium silicates with the aluminum sulfate.

6. An improved method of preparing an aluminum sulfate treated, particulate hydrated calcium silicate product comprising hydrothermally reacting at a temperature of approximately 450°F. stable, hydrothermally formed, high temperature phase hydrated calcium silicates with aluminum sulfate in proportions of approximately 0.20 mols of aluminum sulfate per mol of CaO to effect reaction of approximately 60% by weight of the total CaO component of the hydrated calcium silicates with the aluminum sulfate.

17

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7. An improved method of preparing an aluminum sulfate treated, particulate hydrated calcium silicate product comprising hydrothermally reacting at a temperature of at least about 350°F. stable, hydrothermally formed, high temperature phase hydrated calcium silicates selected from the group consisting of xonotlite and a synthetic hydrated calcium silicate having the composition  $2\text{CaO} \cdot 3\text{SiO}_2 \cdot 1-2.5\text{H}_2\text{O}$  and mixtures thereof with aluminum sulfate in proportions of at least approximately 0.025 mols of aluminum sulfate per mol of CaO to effect reaction of approximately 7% to 60% by weight of the total CaO component of the hydrated calcium silicates with the aluminum sulfate.

8. An improved method of preparing an aluminum sulfate treated, particulate hydrated calcium silicate product comprising hydrothermally reacting at a temperature of approximately 450°F. stable, hydrothermally formed, high temperature phase hydrated calcium silicates selected from the group consisting of xonotlite and a synthetic hydrated calcium silicate having the composition  $2\text{CaO} \cdot 3\text{SiO}_2 \cdot 1-2.5\text{H}_2\text{O}$  and mixtures thereof with aluminum sulfate in proportions of at least approximately 0.025 mols of aluminum sulfate per mol of CaO to effect reaction of approximately 7% to 60% by weight of the total CaO component of the hydrated calcium silicates with the aluminum sulfate.

9. A method of buffering the basic pH characteristics of stable, hydrothermally formed, high temperature phase hydrated calcium silicate products which comprises hydrothermally reacting at a temperature of at least about 350°F.

653797

stable, hydrothermally formed, high temperature phase hydrated calcium silicates with aluminum sulfate in proportions of at least 0.0166 moles of aluminum sulfate per mol of CaO to effect reaction of approximately 7½% by weight of the total CaO component of the hydrated calcium silicates with the aluminum sulfate.

10. A method of buffering the basic pH characteristics of stable, hydrothermally formed, high temperature phase hydrated calcium silicate products which comprises hydrothermally reacting at a temperature of approximately 450°F. stable, hydrothermally formed, high temperature phase hydrated calcium silicates with aluminum sulfate in proportions of at least 0.0166 moles of aluminum sulfate per mol of CaO to effect reaction of approximately 60% by weight of the total CaO component of the hydrated calcium silicates with the aluminum sulfate.

11. A method of buffering the basic pH characteristics of stable, hydrothermally formed, high temperature phase hydrated calcium silicate products which comprises hydrothermally reacting at a temperature of approximately 450°F. stable, hydrothermally formed, high temperature phase hydrated calcium silicates with aluminum sulfate in proportions of at least approximately 0.025 moles of aluminum sulfate per mol of CaO to effect reaction of approximately 7½ to 60% by weight of the total CaO component of the hydrated calcium silicates with the aluminum sulfate.

658797

12. A method of buffering the basic pH characteristics of stable, hydrothermally formed, high temperature phase hydrated calcium silicate products which comprises hydrothermally reacting at a temperature of at least about 350°F. stable, hydrothermally formed, high temperature phase hydrated calcium silicates selected from the group consisting of xonotlite and a synthetic hydrated calcium silicate having the composition  $2\text{CaO} \cdot 3\text{SiO}_2 \cdot 1-2.5\text{H}_2\text{O}$  and mixtures thereof with aluminum sulfate in proportions of at least approximately 0.025 mols of aluminum sulfate per mol of CaO to effect reaction of approximately 7½ to 60% by weight of the total CaO component of the hydrated calcium silicates with aluminum sulfate.

13. A method of buffering the basic pH characteristics of stable, hydrothermally formed, high temperature phase hydrated calcium silicate products which comprises hydrothermally reacting at a temperature of approximately 450°F. stable, hydrothermally formed, high temperature phase hydrated calcium silicates selected from the group consisting of xonotlite and a synthetic hydrated calcium silicate having the composition  $2\text{CaO} \cdot 3\text{SiO}_2 \cdot 1-2.5\text{H}_2\text{O}$  and mixtures thereof with aluminum sulfate in proportions of at least approximately 0.025 mols of aluminum sulfate per mol of CaO to effect reaction of approximately 7½ to 60% by weight of the total CaO component of the hydrated calcium silicates with aluminum sulfate.

653797

14. A particulate hydrated calcium silicate product consisting essentially of stable, hydrothermally formed, high temperature phase hydrated calcium silicates and the hydrothermal reaction product of said high temperature phase hydrated calcium silicates and aluminum sulfate in proportions of at least 0.0166 mole of aluminum sulfate per mol of CaO to effect reaction of at least 5% by weight of the total CaO component of the hydrated calcium silicate with the aluminum sulfate, said product exhibiting an "alum demand" no greater than approximately 4 ml. of an aqueous solution of 0.0216 grams of aluminum sulfate per ml. to achieve a pH of about 5.

15. A particulate hydrated calcium silicate product consisting essentially of stable, hydrothermally formed, high temperature phase hydrated calcium silicates and the hydrothermal reaction product of said high temperature phase hydrated calcium silicates and aluminum sulfate in proportions of at least 0.0166 mole of aluminum sulfate per mol of CaO at a temperature of approximately 450°F. to effect reaction of at least 5% by weight of the total CaO component of the hydrated calcium silicate with the aluminum sulfate, said product exhibiting an "alum demand" no greater than approximately 4 ml. of an aqueous solution of 0.0216 grams of aluminum sulfate per ml. to achieve a pH of about 5.

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21

653797

16. A particulate hydrated calcium silicate product consisting essentially of stable, hydrothermally formed, high temperature phase hydrated calcium silicates and the hydrothermal reaction product of said high temperature phase hydrated calcium silicates and aluminum sulfate in proportions of at least approximately 0.025 mols of aluminum sulfate per mol of CaO at a temperature of at least about 350°F. to effect reaction of approximately 7½ to 60% by weight of the total CaO component of the hydrated calcium silicate with the aluminum sulfate, said product exhibiting an "alum demand" no greater than approximately 4 ml. of an aqueous solution of 0.0216 grams of aluminum sulfate per ml. to achieve a pH of about 5.

17. A particulate hydrated calcium silicate product consisting essentially of stable, hydrothermally formed, high temperature phase hydrated calcium silicates and the hydrothermal reaction product of said high temperature phase hydrated calcium silicates and aluminum sulfate in proportions of at least approximately 0.025 mols of aluminum sulfate per mol of CaO at a temperature of approximately 450°F. to effect reaction of approximately 7½ to 60% by weight of the total CaO component of the hydrated calcium silicate with the aluminum sulfate, said product exhibiting an "alum demand" no greater than approximately 4 ml. of an aqueous solution of 0.0216 grams of aluminum sulfate per ml. to achieve a pH of about 5.

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22

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18. A particulate hydrated calcium silicate product consisting essentially of stable, hydrothermally formed, high temperature phase hydrated calcium silicates selected from the group consisting of xonotlite and a synthetic hydrated calcium silicate having the composition  $2\text{CaO} \cdot 3\text{SiO}_2 \cdot 1-2.5\text{H}_2\text{O}$  and mixtures thereof and the hydrothermal reaction product of the said high temperature phase hydrated calcium silicates and mixtures thereof and aluminum sulfate in proportions of at least approximately 0.025 mols of aluminum sulfate per mol of CaO at a temperature of at least about  $350^\circ\text{F}$ . to effect reaction of approximately 7½ to 60% by weight of the total CaO component of the hydrated calcium silicates with the aluminum sulfate, said product exhibiting an "alum demand" no greater than approximately ¼ ml. of an aqueous solution of 0.0216 grams aluminum sulfate per ml. to achieve a pH of about 5.

19. A particulate hydrated calcium silicate product consisting essentially of stable, hydrothermally formed, high temperature phase hydrated calcium silicates selected from the group consisting of xonotlite and a synthetic hydrated calcium silicate having the composition  $2\text{CaO} \cdot 3\text{SiO}_2 \cdot 1-2.5\text{H}_2\text{O}$  and mixtures thereof and the hydrothermal reaction product of the said high temperature phase hydrated calcium silicates and mixtures thereof and aluminum sulfate in proportions of at least approximately 0.025 mols of aluminum sulfate per mol of CaO at a temperature of approximately  $450^\circ\text{F}$ . to effect reaction of approximately

A

23

658797

7½ to 60% by weight of the total CaO component of the hydrated calcium silicates with the aluminum sulfate, said product exhibiting an "alum demand" no greater than approximately 4 ml. of an aqueous solution of 0.0216 grams aluminum sulfate per ml. to achieve a pH of about 5.